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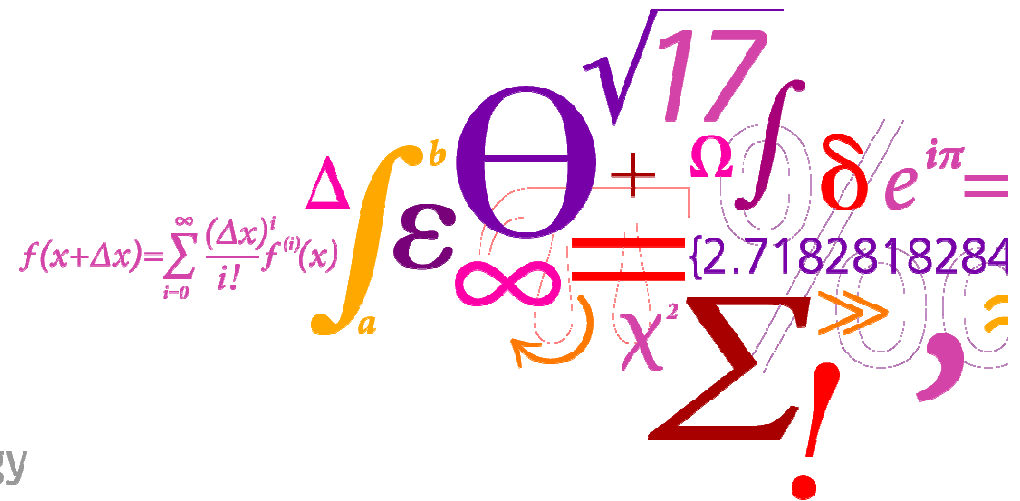
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Measuring the elastic strain of individual grains in a polycrystalline material

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Measuring the elastic strain of individual grains in a polycrystalline material

- Why?
 - To study grain-grain interactions in deformed materials – Does the strain depend on the grain orientation and/or the neighbouring grains?
 - To study residual stresses
 - To study crack formation and propagation and the role of reinforcements for the process
- How?
 - Farfield 3DXRD
- July 2008 workshop on 3DXRD software for strain in grains:
 - C. Aydiner, J. Bernier, J. Wright, U. Lienert, P. Reischig (W. Ludwig)
 - M. Miller, A. Borbely
- FitAllB – Fable package for fitting grain resolved centre of mass positions, orientations and elastic strains

FitAllB

$$\sum_{i,j(i)} \left(\Gamma_{ij}^{-1} \bar{G}_{ij} - \frac{\lambda}{2\pi} U_i B_i \bar{G}_{hkl,ij} \right)^T V_{ij}^{-1} \left(\Gamma_{ij}^{-1} \bar{G}_{ij} - \frac{\lambda}{2\pi} U_i B_i \bar{G}_{hkl,ij} \right)$$

Observations
3 per reflection

$$\bar{G}_{ij} = \left[\frac{\bar{d}_{ij}}{|\bar{d}_{ij}|} - \begin{pmatrix} 1 \\ 0 \\ 0 \end{pmatrix} \right]$$

Global parameters
10

Grain parameters
12 per grain

$$\bar{d}_{ij} = R \begin{pmatrix} 0 \\ y_{det,ij} - y_{det,0} \\ z_{det,ij} - z_{det,0} \end{pmatrix} + \begin{pmatrix} D \\ 0 \\ 0 \end{pmatrix} - \Gamma_{ij} \begin{pmatrix} x_{0,i} \\ y_{0,i} \\ z_{0,i} \end{pmatrix}$$

Fable input

- peaksearch:
 - images →
 - filtered peaks file (.flt: ω , dety, detz, spotid, intensity)
- transformation:
 - peak positions (.flt) + detector parameters (.par) →
 - scattering g-vectors (.gve)
- GrainSpotter:
 - g-vectors →
 - oriented grains (.log: spotid, h, k, l, orientations, positions)

FitAllB input

- `flt_file` `al20_peaks_t25.flt`
- `par_file` `al20_detector.par`
- `log_file` `al20_grainspotter.log`
- `structure_file` `al.cif`
- `dety_size` 2048
- `detz_size` 2048
- `w_step` 0.5
- `w_limit` -22.5 22.5 67.5 112.5
- `crystal_system` cubic
- `c11` 10.8e10
- `c12` 6.22e10
- `c44` 2.84e10
- `skip` 4 # skip grain 4 in `al20_grainspotter.log`
- `ia` 0.2
- `min_refl` 60

FitAllB input

- w 0 # Fit omega stage tilt parameter wy (wedge)
- center 0 # Fit beam centre on detector in y direction, cy
- pixel 0 # Fit pixel size py and pz
- tilt 0 # Fit detector tilt parameters tx, ty, tx
- L 0 # Fit sample-to-detector distance

- rod 1 # Fit orientations (Rodrigues vector)
- xyz 1 # Fit positions
- eps 1 # Fit strain tensors

FitAllB output

- A parameter file containing the following parameters for each grain:
 - grainno mean_IA grainvolume x y z
 - rodx rody rodz U11 U12 U13 U21 U22 U23 U31 U32 U33
 - eps11 eps22 eps33 eps23 eps13 eps12
 - eps11_s eps22_s eps33_s eps23_s eps13_s eps12_s
 - sig11 sig22 sig33 sig23 sig13 sig12
 - sig11_s sig22_s sig33_s sig23_s sig13_s sig12_s
- An error file containing the estimated errors of the above parameters

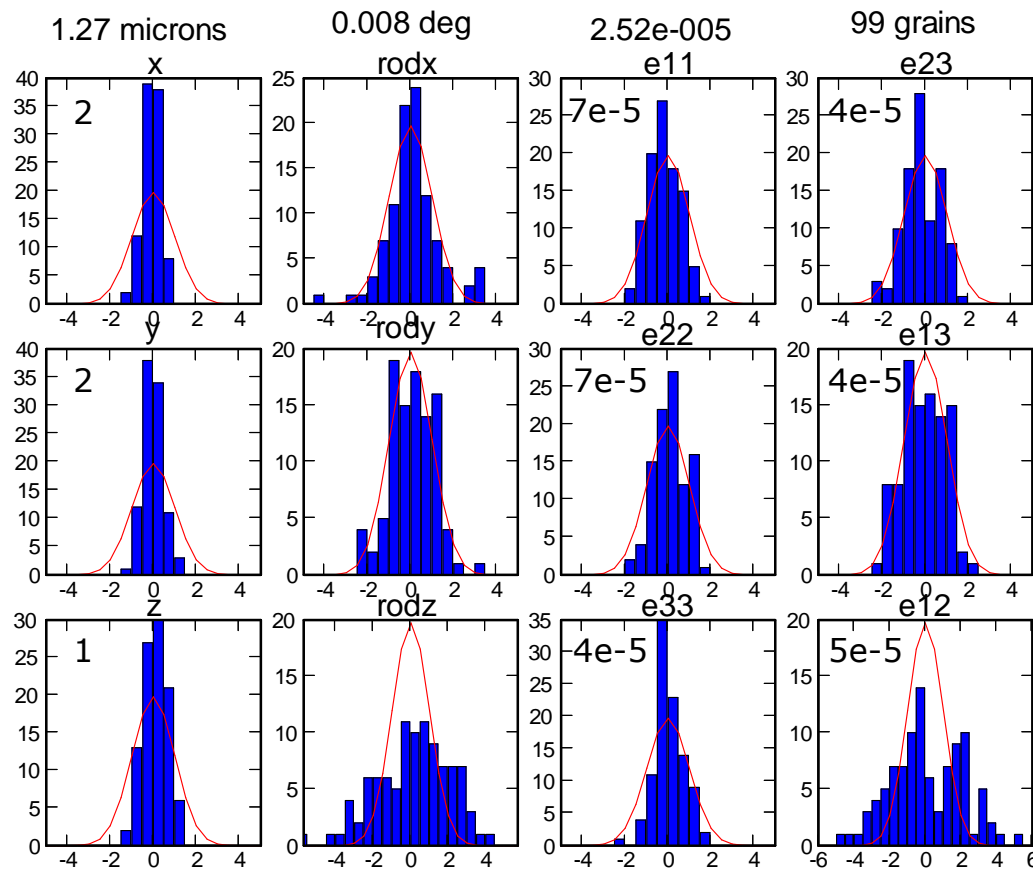
Tests on simulated data - PolyXSim

- Model systems
 - F.C.C. or B.C.C. metals
 - 100-200 illuminated grains
 - Positions randomly distributed in cylinder with $\varnothing=0.5-1$ mm and $h=0.01-0.1$ mm
 - Random orientations
 - Lognormal distribution of grain sizes
 - Random strain, Gaussian distribution with $\mu=0$ and $\sigma=0.001$
 - ~ 70 keV, 2048×2048 pixels detector, 50×50 μm pixels (ID11 Frelon4M) sample-to-detector distance to give 5 full diffraction rings
- Present example
 - 100 grains of IF steel (B.C.C)
 - ω -ranges: $-22.5 \rightarrow 22.5^\circ$ and $67.5 \rightarrow 112.5^\circ$ in steps of 0.5°

Simulated data and error estimation

Idealised geometry

Detector discretisation



For each of the 12 grain parameters:

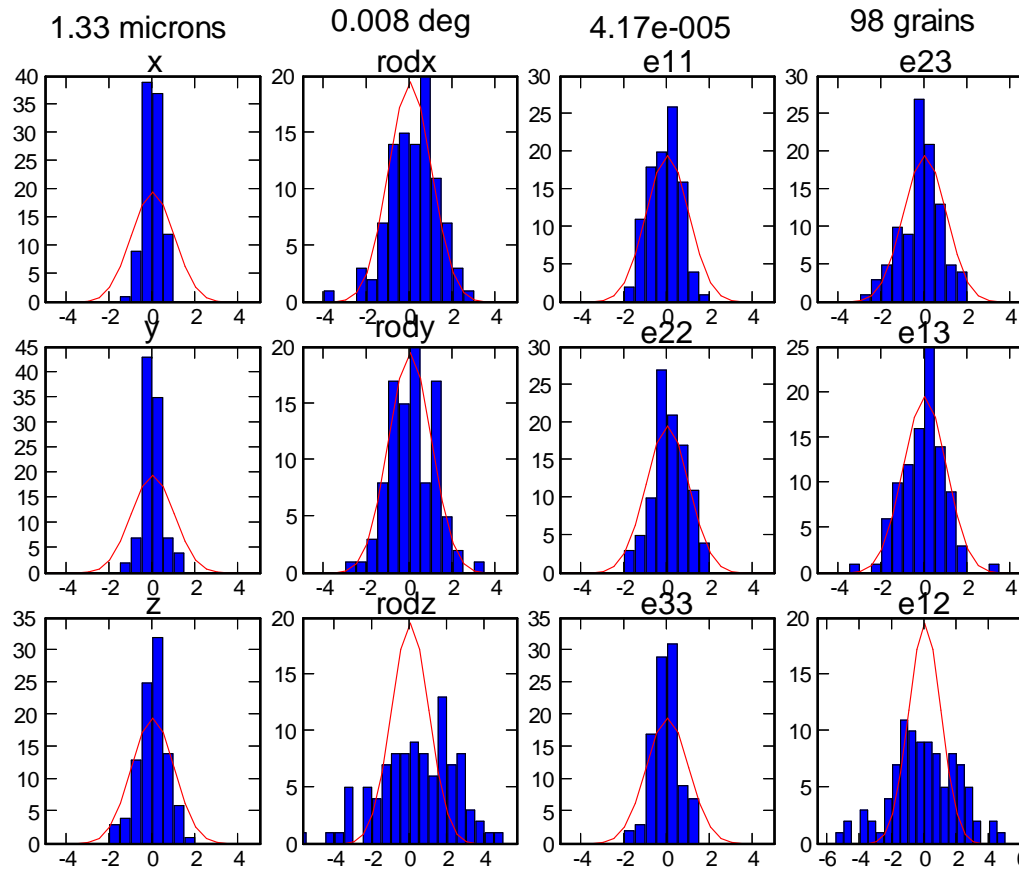
x-axis:
(refined – true value)/
estimated error

y-axis:
Number of observations

Red curve:
Gaussian with $\mu=0$ and $\sigma=1$, expected for correct error estimation

Idealised geometry

Diffractometer vibrations in y and z (2D-Gaussian, $\sigma=1 \mu\text{m}$)



For comparison:

1.27 microns
0.008 deg
2.52e-5

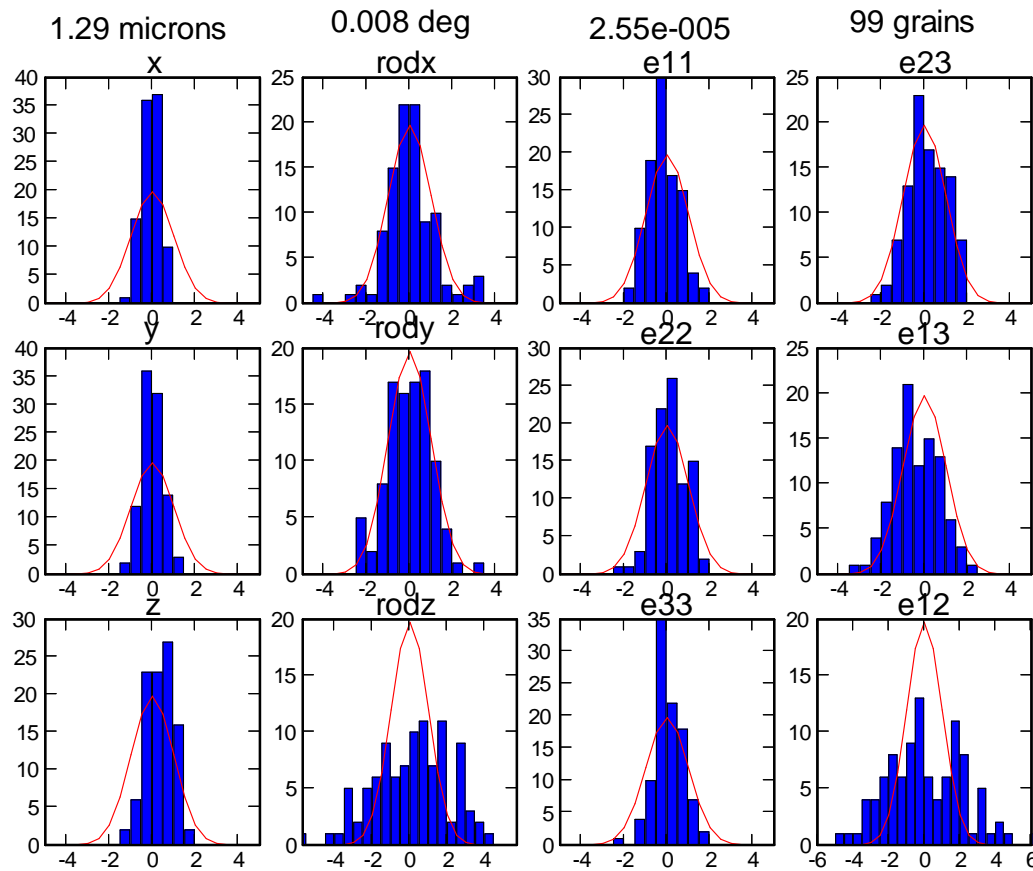
Fitting global parameters

- Fitglobalgrain or Fitglobal for multigrain global parameter refinements
- Same input file as for FitAllB, but with the following options:

- w 1 # Fit omega stage tilt parameter wy (wedge)
- center 1 # Fit beam centre on detector in y direction, cy
- pixel 0 # Fit pixel size py and pz
- tilt 1 # Fit detector tilt parameters tx, ty, tx
- L 1 # Fit sample-to-detector distance

- rod 1 # Fit orientations (Rodrigues vector)
- xyz 1 # Fit positions
- eps 0 # Fit strain tensors

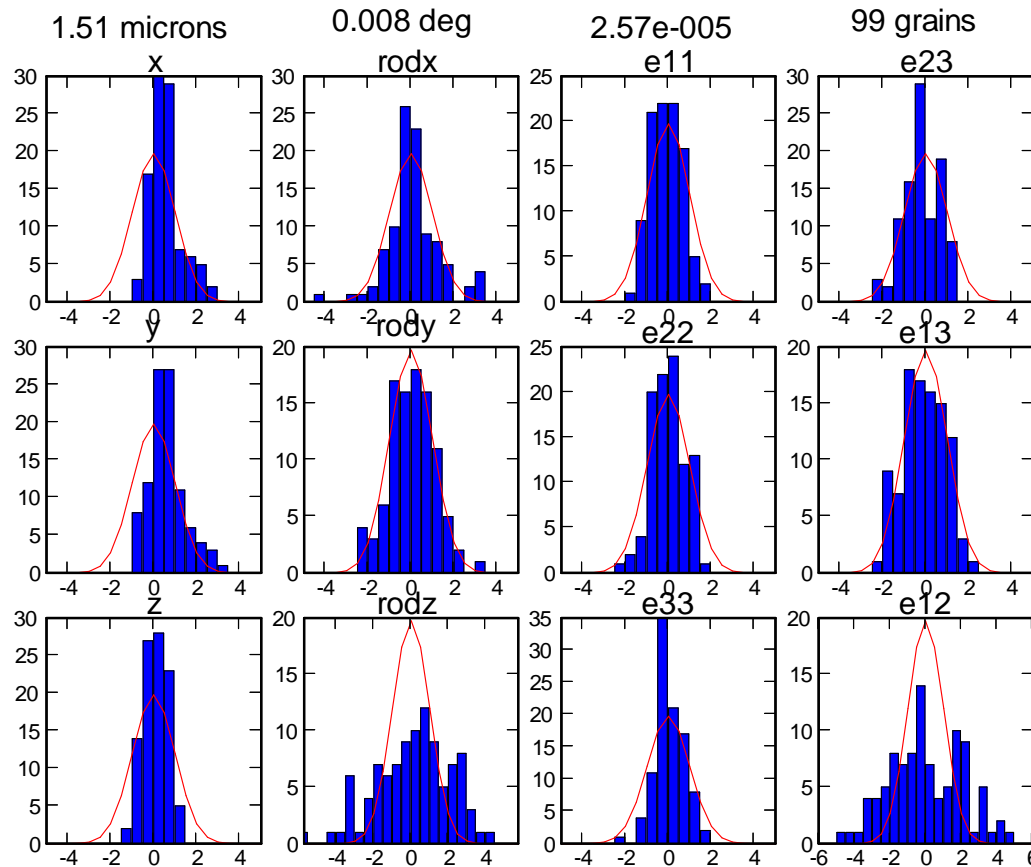
Sample tilt (wedge) off by 0.001°



For comparison:

1.27 microns
0.008 deg
2.52e-5

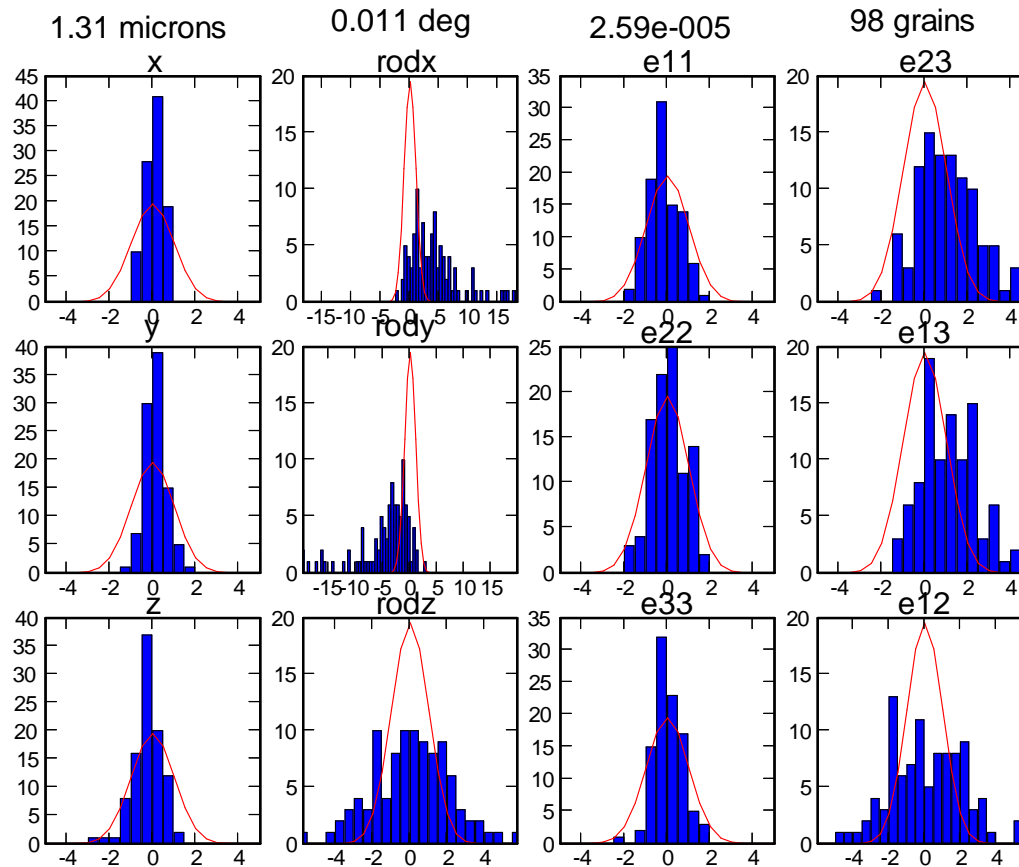
Beam centre on detector off by 0.01 pixels in y direction (0.5 μm)



For comparison:

1.27 microns
0.008 deg
2.52e-5

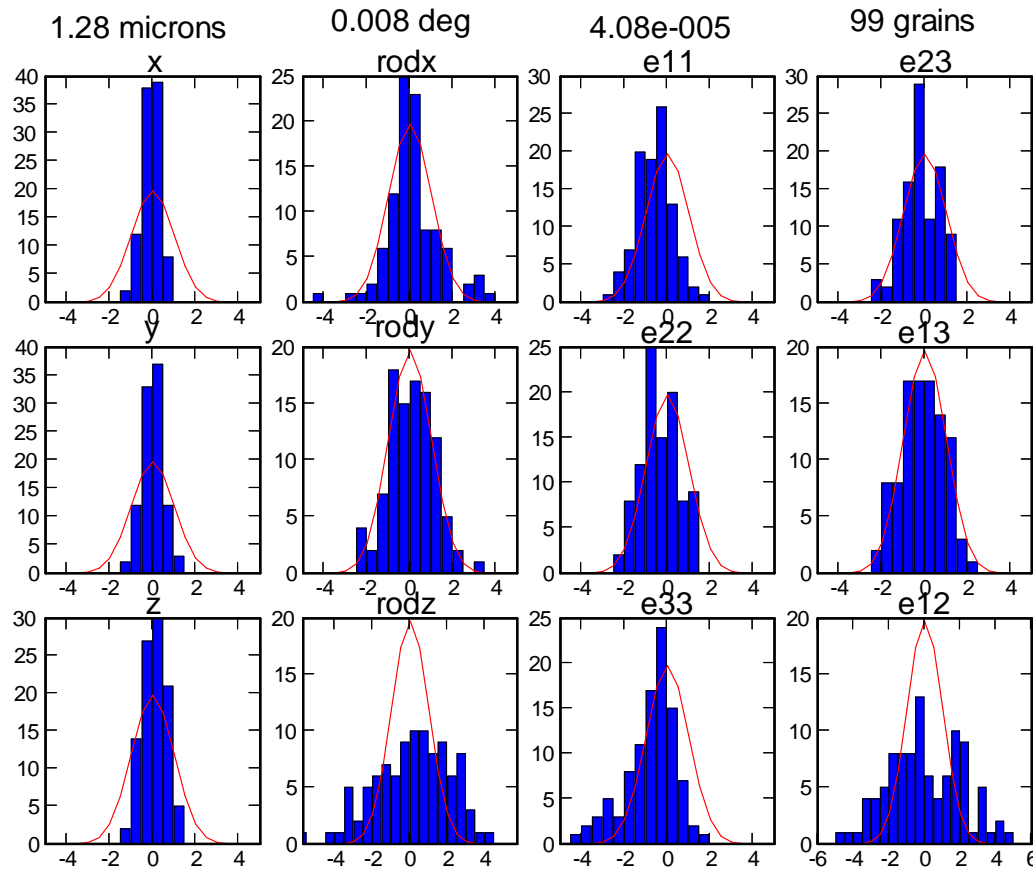
Detector tilt off by 0.006°



For comparison:

1.27 microns
0.008 deg
 2.52×10^{-5}

Sample-to-detector distance off by 2 μm on 200 mm ($1\text{e-}5$)



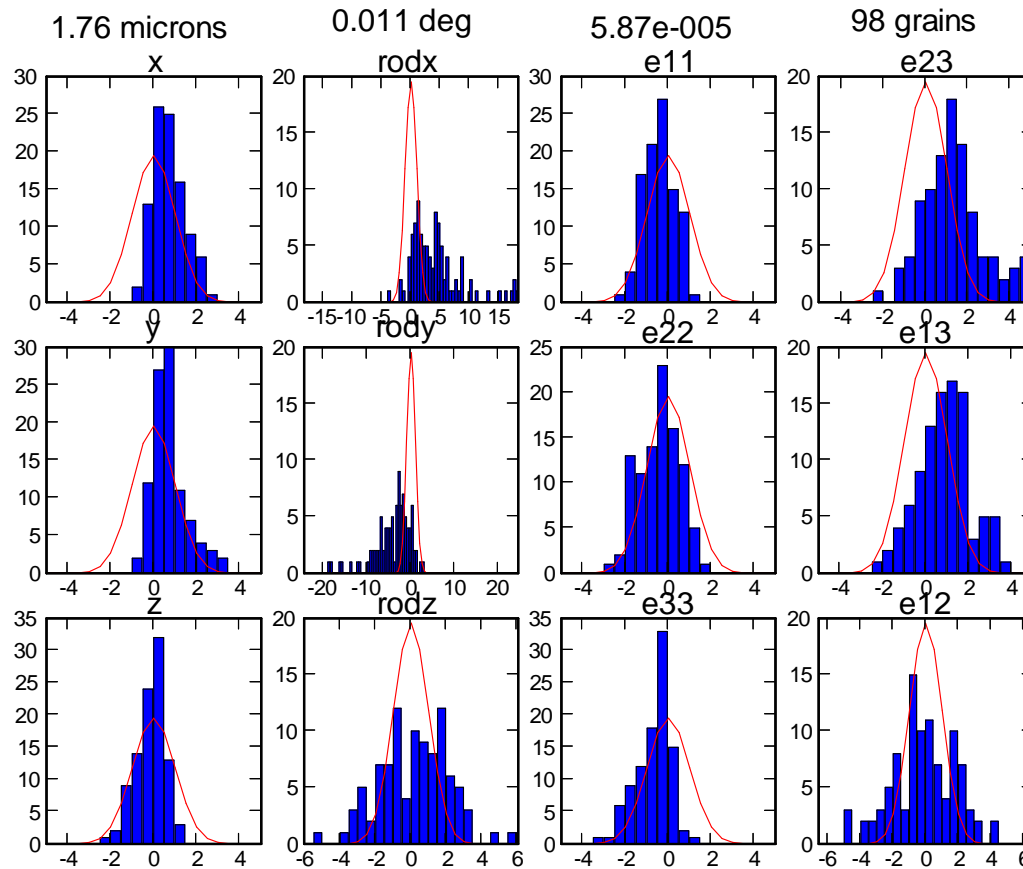
For comparison:

1.27 microns
0.008 deg
2.52e-5

Fitted geometry

Detector discretisation

Diffractometer vibrations



For comparison:

1.27 microns
0.008 deg
2.52e-5

With vibrations:

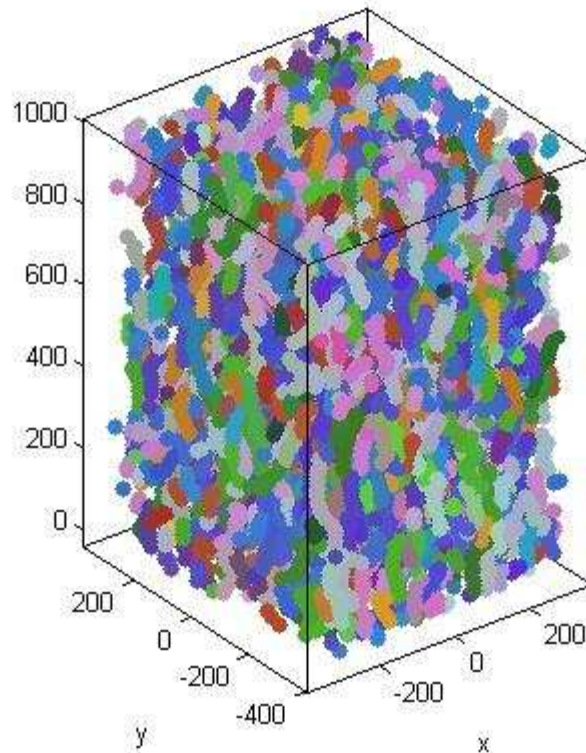
1.33 microns
0.008 deg
4.17e-5

Experimental data

Refining global parameters

- Global parameters for Al reference single crystal
 - wedge = -0.0206°
 - y_center = 1019.06
 - tilt_x = -0.00041
 - tilt_y = 0.00303
 - tilt_z = -0.01337
 - L = 252.355 mm
 - Possible to index $\sim 35\%$ of the reflections
- Global parameters for 5 layers of undeformed Cu, 161 grains
 - wedge = $-0.3552(7)^\circ$
 - y_center = 1018.743(11)
 - tilt_x = $-0.00066(1)$
 - tilt_y = $0.00414(10)$
 - tilt_z = $-0.01063(10)$
 - L = 252.358(4) mm
 - Possible to index $\sim 45\%$ of the reflections

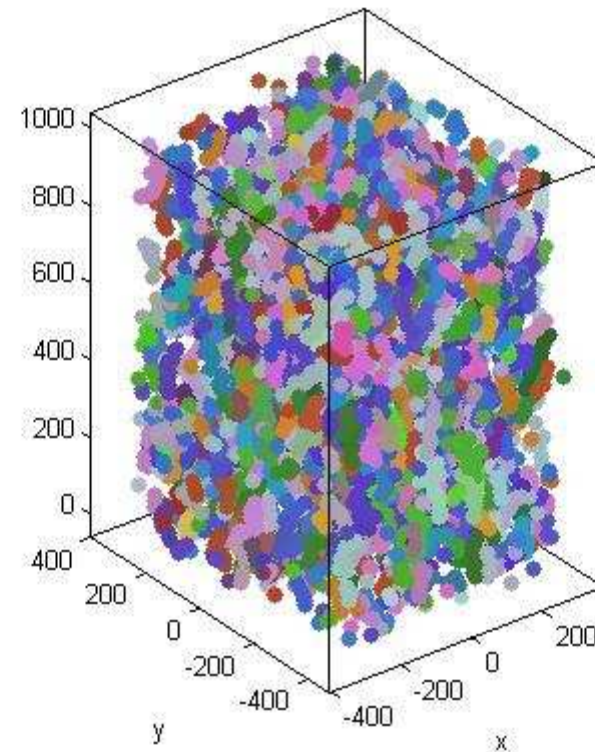
Experimental data, level 1: Centre of mass position and orientations IF steel, ex situ deformed



Undeformed
1939 grains

Est. error on pos.: 3 μm

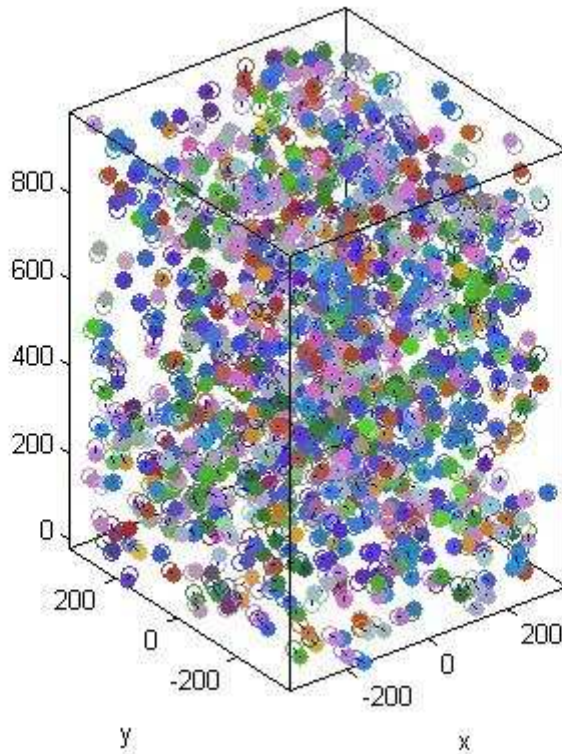
NB! Position fit using near-field data



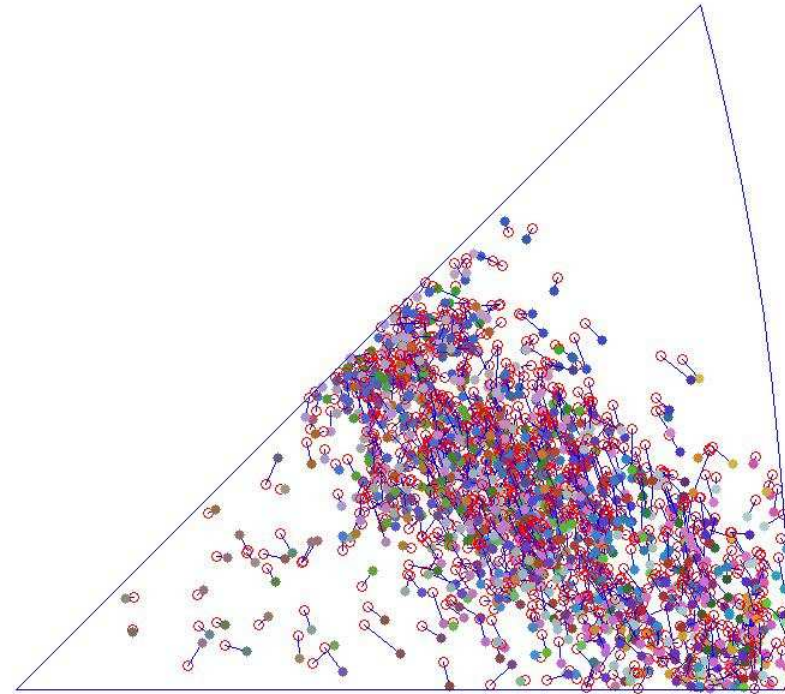
3% deformed
1766 grains
5 μm

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Experimental data, level 1: Centre of mass positions and orientations IF steel, ex situ deformed



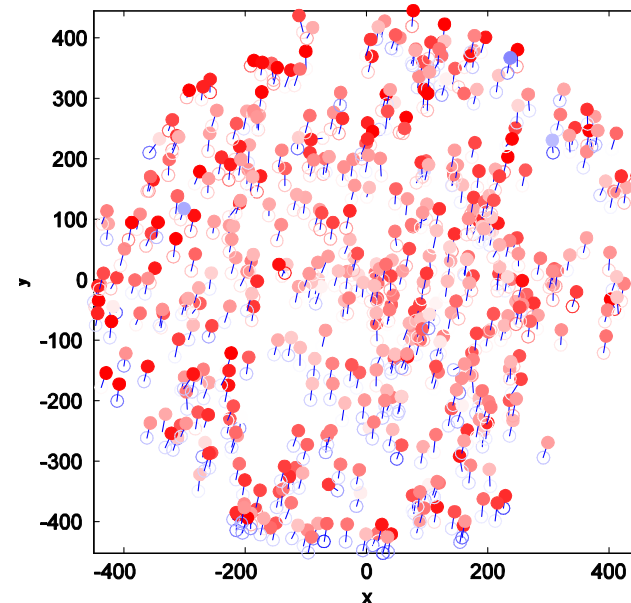
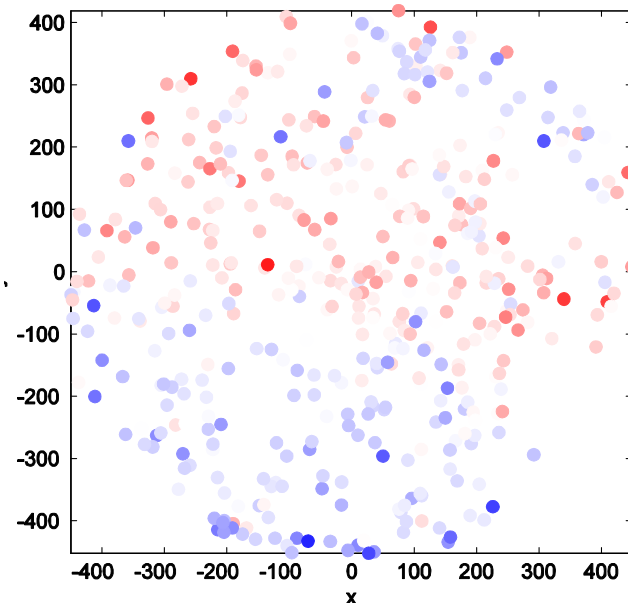
Possible to match 1186 grains
(of 1939 and 1766)



To study grain rotations during
deformation

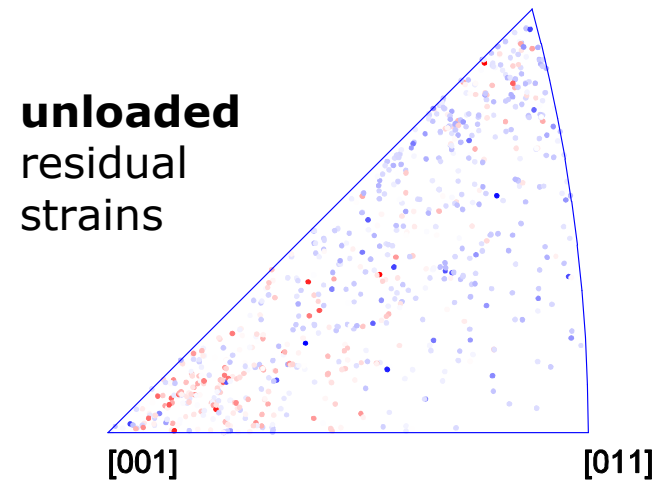
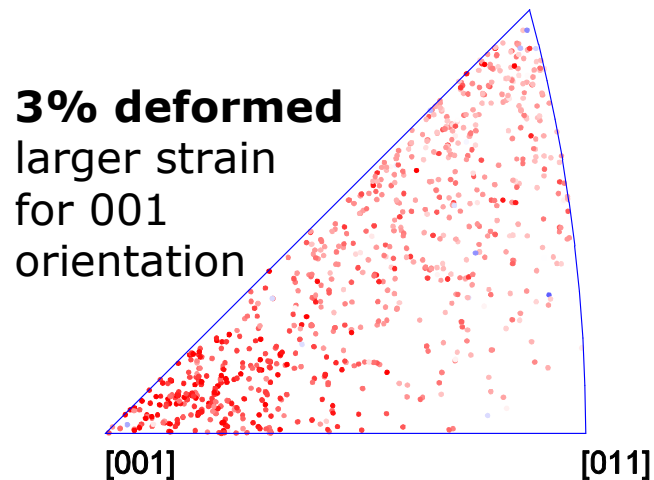
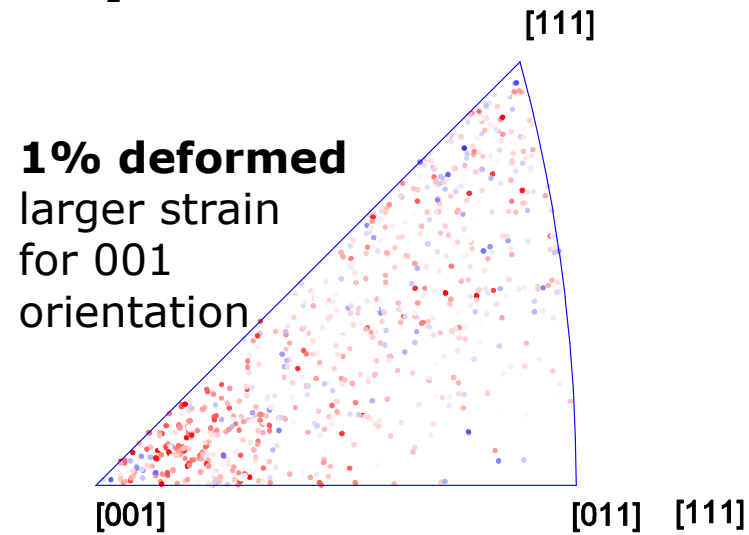
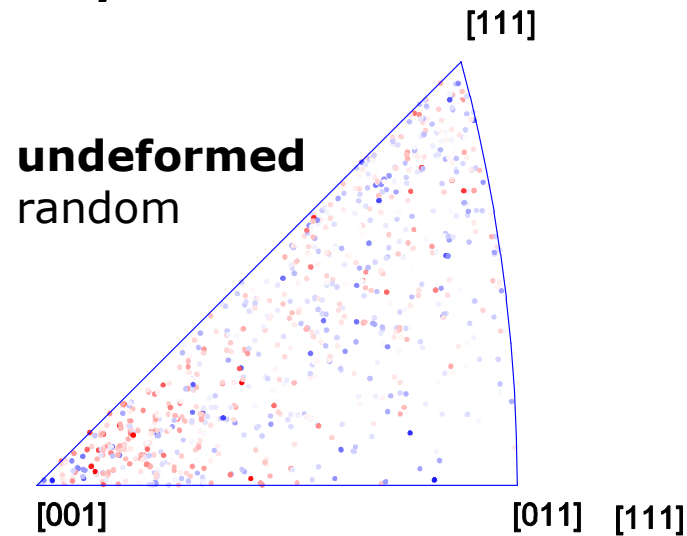
Experimental data, level 2: Positions, orientations and strains Cu, deformed tensionally in situ

- Strain levels: undeformed, 1%, 3% and unloaded
- Sample diameter 1 mm, 5 layers of 0.1 mm mapped
- 800 large grains indexed and refined
- 450 of these match between undeformed and 3% deformation



J. Oddershede, G. Winther, H.F. Poulsen, L. Margulies, M. Moscicki, S. Schmidt, J. Wright – in progress

Experimental data, level 2a: Correlating strain and orientation Cu, deformed tensionally in situ



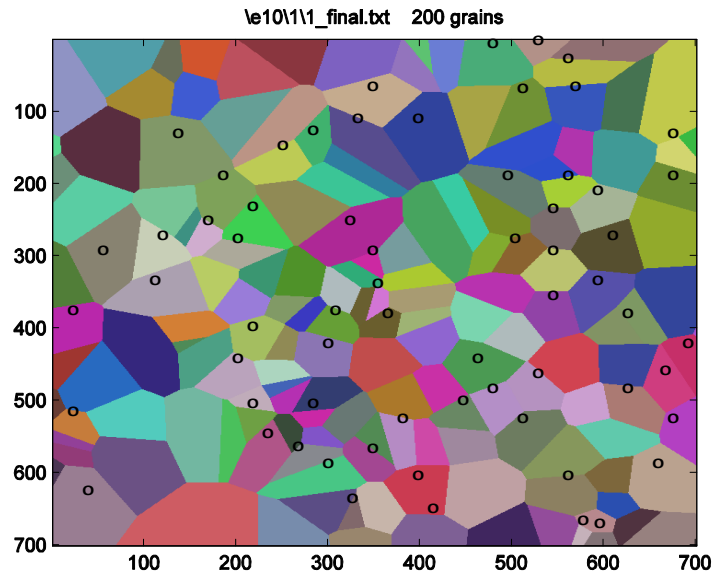
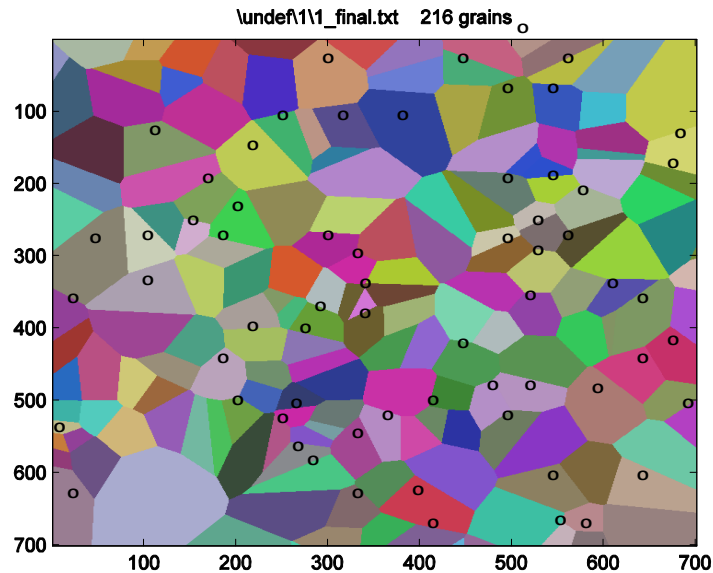
Experimental data, level 3: Laguerre tessellation, grain maps and grain interaction studies

- Laguerre tessellation:
 - Method to get 3D grain map from centre of mass positions and relative grain volumes
- Test on position, volumes and grain shapes from from microtomography on meta-stable beta-titanium alloy
(A. Lyckegaard, E.M. Lauridsen, W. Ludwig, R.W. Fonda, H.F. Poulsen)

	Voronoi	Laguerre					
Error type	None	None	Volume	CMS			
Std. of error, 3 sigma	-	-	10%	2 μm	4 μm	7 μm	10 μm
% Correct labelled voxels	59.72	86.30	86.26	85.88	84.72	81.85	78.25
% grains with all neighbours correct	7.82	31.75	30.90	28.80	23.82	16.99	10.15
# erroneously extra neighbours/grain	1.87	0.58	0.59	0.62	0.73	0.93	1.23
# erroneously missing neighbours/grain	1.29	0.64	0.65	0.69	0.76	0.96	1.24
# total of wrong neighbours/grain	3.16	1.22	1.24	1.31	1.49	1.89	2.47

Table 1: Average similarity measures for the tessellations: Voronoi (N=1), Laguerre without errors (N=1), Laguerre with 10% volume errors (N=17) and Laguerre with 2 μm , 4 μm , 7 μm and 10 μm CMS errors (N=17).

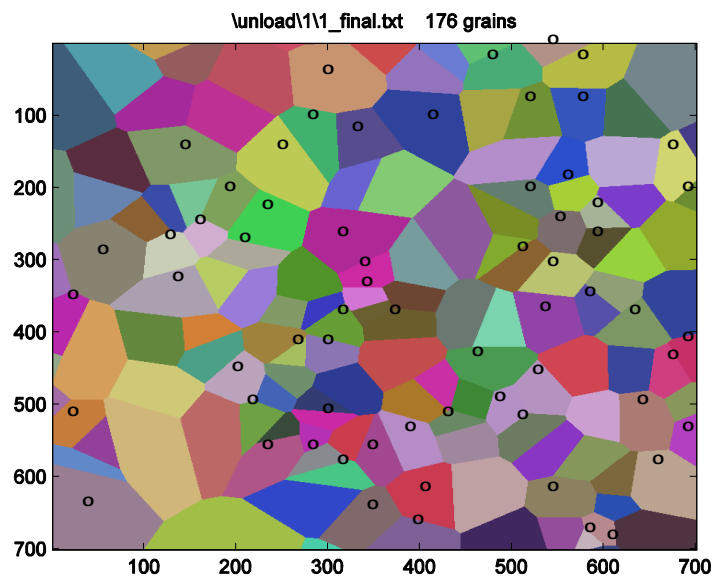
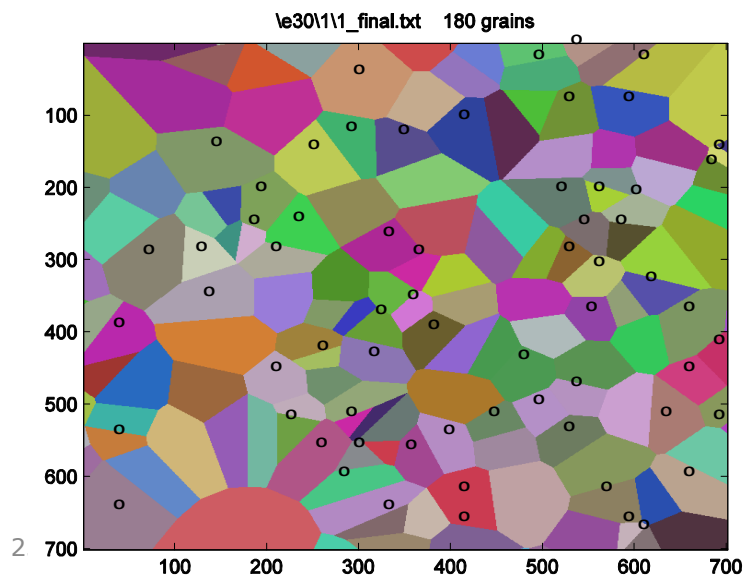
Experimental data, level 3: Cu, deformed tensionally in situ



estimated error
on position:
10 μm

62 grains
visually
matched at all
strain levels

Sample slip/
elongation



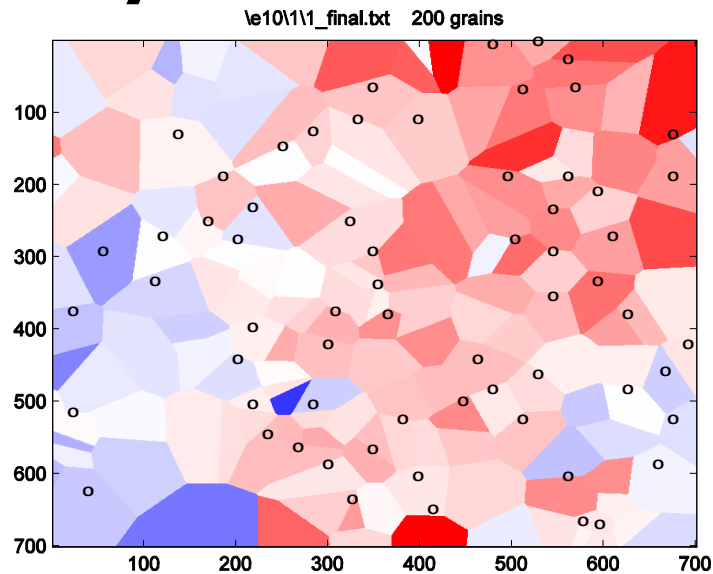
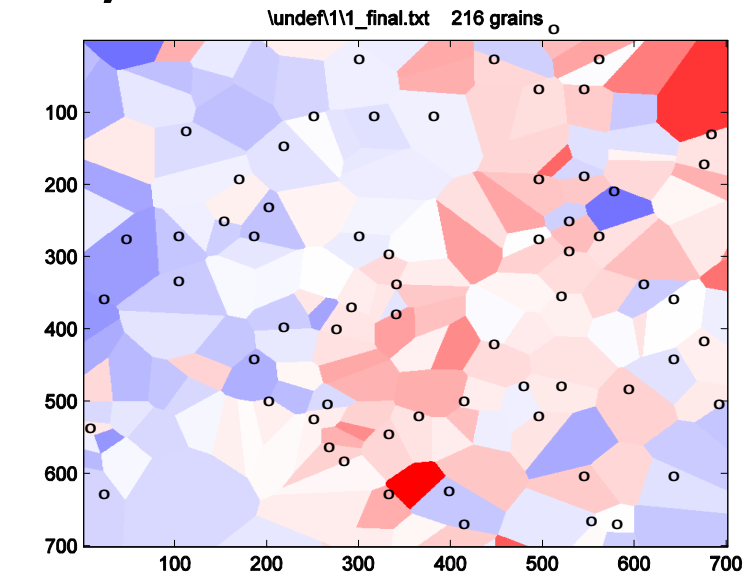
Twins!!

Combine
knowledge
from adjacent
layers/strains
to improve
incomplete
indexing

2-apr-2009

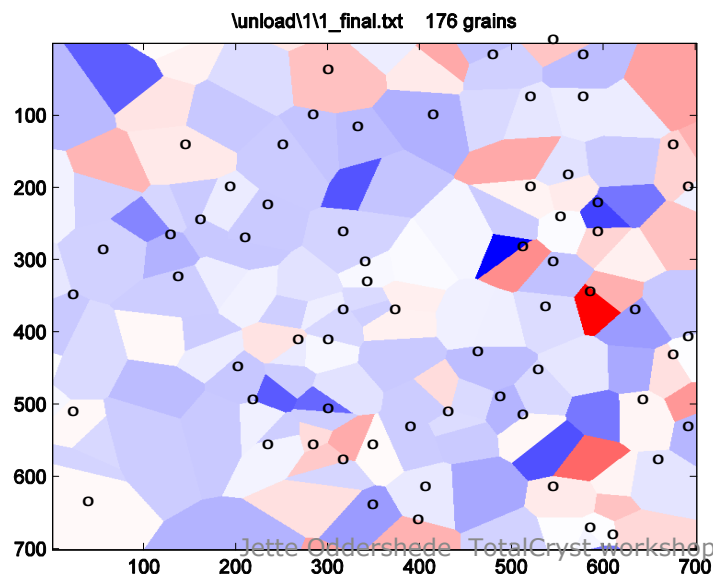
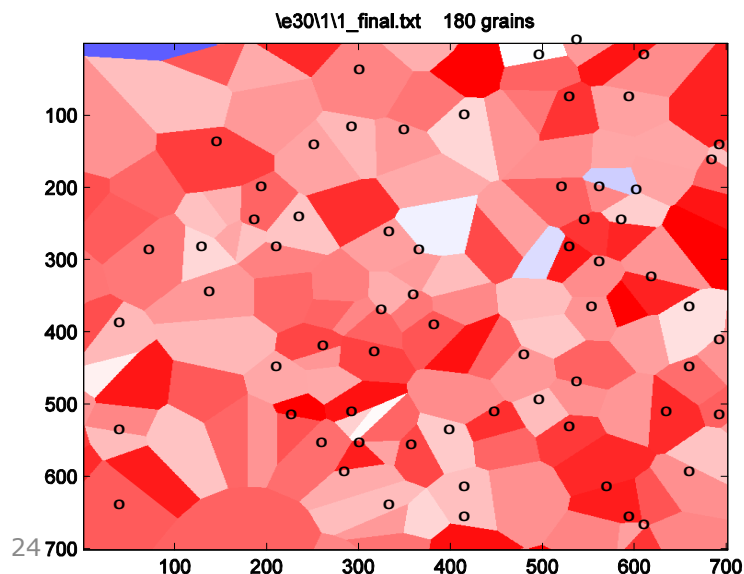
Experimental data, level 3

Cu, deformed tensionally in situ



undeformed
 $\epsilon_{33}^{avg} = 1.2e-5$
 $\epsilon_{33}^{err} = 1.1e-4$

1%
 $\epsilon_{33}^{avg} = 1.6e-4$
 $\epsilon_{33}^{err} = 1.1e-4$



3%
 $\epsilon_{33}^{avg} = 5.4e-4$
 $\epsilon_{33}^{err} = 1.2e-4$

unloaded
 $\epsilon_{33}^{avg} = -5.3e-5$
 $\epsilon_{33}^{err} = 1.2e-4$

Conclusions

- FitAllB for refining centre of mass grain positions, orientations and strain tensors and Fitglobalgrain/Fitglobal for refining global experimental parameters
- Simulated data used to validate error estimation and illustrate the necessity for accurate global parameters
- IF steel ex situ
 - ~2000 grains, good statistics
 - The use of near-field data for position fit significantly improved these
- Cu in situ
 - Average estimated error on ϵ_{33} strain in tensile direction $\leq 1.2e-4$
 - Strain evolution along tensile axis detected
 - Orientation dependence of ϵ_{33} detected

Outlook

- IF steel ex situ:
 - Too low percentage of grains matched between undeformed and 3% deformed, must be improved
 - 6% and 9% data
- Cu in situ:
 - Twins, indexing
 - Strain and correction for spatial distortion of detector
 - Analysis of data measured at 0.2 % and 0.4 % deformation
 - Present data measured for ω -ranges: $-150 \rightarrow -30^\circ$ and $30 \rightarrow 150^\circ$ in steps of 0.25° . Is one ω -range enough (speed gain)?
 - One layer remeasured in steps of 0.1° , is this an improvement?
- APS beamtime application for studying the grain resolved stress evolution around crack tips.

Acknowledgements

- J. Wright & H.O. Sørensen, my python and fable gurus
- G. Winther, everything on deformation theory
- Everyone who helped prepare the samples and collect the data
- M. Moscicki and A. Borbely, MPIE Düsseldorf, stress rig

Motivation

- Goal:
 - To determine the centre-of-mass elastic strains (and stresses - type II) in many (100+) grains to an accuracy of 10^{-4}
- To study what:
 - grain-grain interactions in elastically deformed materials
 - crack formation and propagation
 - residual stresses
- Approach
 - FitAllB – Fable package for fitting grain resolved centre of mass positions, orientations and elastic strains

Potential problems 2: Peak overlap

- Especially for textured and/or deformed materials
- Solutions:
 - Illuminate a smaller volume
 - Filter out peaks covering more than a certain number of pixels
 - Use several thresholds in peaksearch and merge the outcome